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THE INFLUENCE OF ALOE VERA CONCENTRATION ON MORPHOLOGY AND TENSILE PROPERTIES OF ELECTROSPUN ALOE VERA-PVA NANOFIBER

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ABSTRACT

THE INFLUENCE OF ALOE VERA CONCENTRATION ON MORPHOLOGY AND TENSILE PROPERTIES OF ELECTROSPUN ALOE VERA-PVA NANOFIBER. Aloe Vera (AV)-poly (vinyl alcohol) (PVA) electrospinning solution with 0%, 2%, 4% and 6% AV concentrations were electrospun to explore the influence of AV concentration on the morphology and tensile properties of the nanofibers membranes. The results showed that the change in the morphology and tensile properties of electrospun AV-PVA nanofibers depends upon AV concentration of electrospinning solution. Mean fiber diameter at 2% (342 ± 0.8 nm) and 4% (337 ± 0.6 nm) AV concentrations is smaller than that at 0% (377 ± 1.1 nm) and 6% (810 ± 3.4 nm) AV concentrations with relatively uniform size distribution. Tensile strength and modulus at AV concentration of 2% (5.74 MPa and 33.99 MPa) and 4% (6.38 MPa and 34.75 MPa) are higher than that at 0% (4.98 MPa and 26.45 MPa) and 6% (2.22 MPa and 13.69 MPa), respectively. AV-PVA electrospinning solution with AV concentration ranging from 2% to 4% is considered being the optimum range that could produce the electrospun nanofibers with the tensile properties included in the range of that specified by the native skin.

Keywords: Aloe vera, PVA, Electrospinning, Nanofiber, Tensile properties

ABSTRAK

PENGARUH KONSENTRASI *OLOE VERA* PADA MORFOLOGI DAN SIFAT TARIK MEMBRAN NANOFIBER *ALOE VERA-PVA*. Larutan *Aloe vera (AV)-poly (vinyl alcohol) (PVA)* sebagai larutan electrospinning dengan konsentrasi *AV* 0%, 2%, 4% dan 6% dipabrikasi dengan metode *electrospinning* untuk mengetahui pengaruh variasi konsentrasi *AV* terhadap morfologi dan sifat tarik membran nanofiber *AV-PVA*. Hasil penelitian menunjukkan bahwa variasi konsentrasi *AV* pada larutan electrospinning menyebabkan perubahan morfologi dan sifat tarik membran nanofiber *AV-PVA*. Diameter fiber rata-rata pada membran dengan konsentrasi *AV* 2% (342 ± 0,8 nm) dan 4% (337 ± 0,6 nm) lebih kecil disbanding 0% (377 ± 1,1 nm) dan 6% (810 ± 3,4 nm) disertai distribusi ukuran fiber yang relatif homogen. Selain itu, kuat tarik dan modulus elastisitas tarik pada membran dengan konsentrasi *AV* 2% dan 4% masing-masing (5,74 MPa and 33,99 MPa) dan (6,38 MPa and 34,75 MPa) juga menunjukkan nilai yang lebih tinggi dari pada membran 0% (4,98 MPa and 26,45 MPa) dan 6% (2,22 MPa and 13,69 MPa). Larutan electrospinning *AV-PVA* dengan rentang konsentrasi *AV* 2% hingga 4% dapat disimpulkan sebagai rentang optimum pada pembuatan membran nanofiber *AV-PVA* karena sifat tarik membran yang dihasilkan termasuk dalam rentang sifat tarik kulit alami manusia.

Kata kunci: *Aloe vera*, *PVA*, *Electrospinning*, Nanofiber, Sifat tarik

INTRODUCTION

Aloe Vera (AV) is a kind of plant that is easily cultivated and abundantly available in tropical climate regions. AV that has been known since 4th century is also called a wonder plant [1] because it has various beneficial compounds being potentially used for wide ranges applications such as foods, beverages, cosmetic and healthcare. AV plant and cross-section of AV leaf (Figure 1) show that the inner gel (leaf gel) is made of 99% of water and the residual components are essential amino acid, glucomannans, minerals, lipids, vitamins, polysaccharides, major polypeptides, proteins and antioxidants [2]. The best medicinal properties contained in AV such as antimicrobial, anticancer and antidiabetic [3,4], leads to increasing research interest of AV as a natural biomaterial. Research toward nanotechnology-based AV has been recently developed, especially in biomedical applications such as wound healing, wound dressing, drug delivery, and tissue engineering.

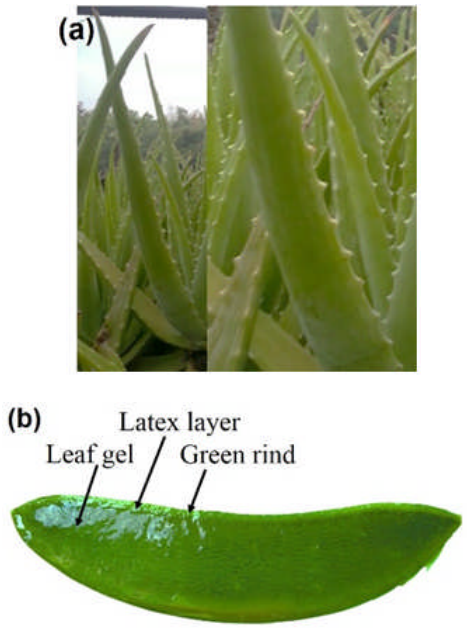

Figure 1. (a). AV plant and (b). cross-section of AV leaf.

Some studies on preparation and characterization of nanofiber membranes based AV for wound healing wound dressing and tissue engineering applications have been carried out. Miquel and co-workers have produced an asymmetric membrane using the electrospinning method [5]. The membrane consisted of two distinct layers: i.e. a bottom and a top layers. The layer made of blended chitosan and AV was positioned in the bottom layer due to their antimicrobial property [4, 6] for enhancing the wound healing process, but polycaprolactone (PCL) was set in the top layer. The mechanical properties of those membranes showed tensile properties being matched with the standard value categorized by the native skin: i.e., Young modulus (4.6 - 20 MPa), tensile strength (5 - 30 MPa) and elongation-to-break (35 - 115%) [5,7]. Besides, electrospun nanofiber of PCL/AV has also been studied by varying ratio of PCL/AV from 100/0 to 70/30 for tissue engineering application [8]. PCL/AV at the ratio of 70/30 with uniform fiber diameter showed in improving cellular compatibility of the cell on nanofiber membrane in which the role of AV can provide sufficient support for cell growth. Besides, blended chitosan and AV solution as a spinning solution has been used for fabricating the nanofiber membranes [9]. In this case, chitosan was liquefied in a various concentration of acetic acid from 50% to 90%. The result indicated that the best fiber structure with an average fiber diameter of 183 nm formed at the acetic acid concentration of 90%. Also, the electrospun nanofiber membrane made of PVA/AV with 5% (w/v) concentration showed average fiber size smaller than that of a neat PVA [10], indicating that PVA and AV are compatible and they can be mixed homogeneously. Electrospun AV/hybrid polymers (PVA/polyvinyl pyrrolidone (PVP)/polyethylene glycol (PEG)) nanofibers with varying AV concentrations of 1, 2 and 3% also designated that the addition of AV reduces the size of formed fibers and shows beads-free fiber structure [11] as reported in Ref. [10].

Studies on electrospun nanofiber-based AV have mostly investigated the characterization of fiber morphology related to the solution concentration. However, a limited number of research discussing the mechanical properties has been reported, especially investigation on electrospun AV-PVA nanofibers. In the electrospinning process, solution viscosity is the function of solution concentration which plays a significant role in inducing the morphology and tensile properties of produced electrospun nanofibers [12-15].

In this our preliminary study, the AV-PVA nanofiber membranes were prepared by the electrospinning technique with the expected tensile properties of the membrane included in the range of the properties indicated by the native skin. Therefore, the objectives of this study are to investigate the effect of AV-PVA solution with different AV concentrations on morphology and tensile properties of electrospun AV-PVA nanofibers.

EXPERIMENTAL METHODS

Materials and Fabrication of the Nanofiber Membranes

AV and PVA used in this work were commercial extract AV powder and PVA Gohsenol (PVOH/PVA, Mw: 22000 g/mol), respectively. PVA solution (10% w/v) was prepared by dissolving 10 g PVA into 100 ml distilled water at 80°C for an hour with continuous magnetic stirring. Various AV concentrations in AV/PVA solution (0, 2, 4 and 6 %) were prepared by mixing AV powder in

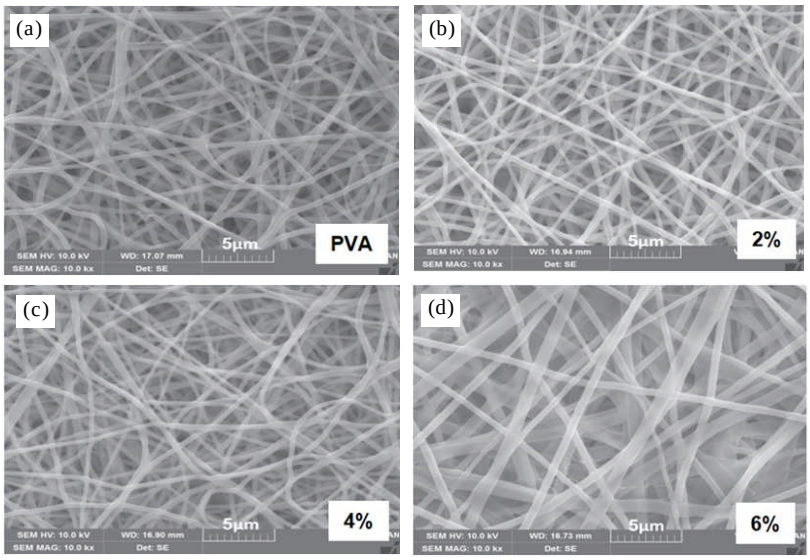

Figure 2. SEM micrographs of AV/PVA nanofiber membranes with differences in concentration of AV/PVA (a). 0% (neat PVA), (b). 2%, (c). 4% and (d). 6% (w/v).

PVA (10% w/v) and stirred at room temperature for 2 h. The viscosity of each concentration of AV-PVA solution was measured with a viscometer (Brookfield Viscometer).

The electrospinning solution of AV-PVA was subsequently fabricated to be the nanofiber membranes by electrospinning technique operating under the optimized conditions, *viz*. Applied DC voltage 10 kV, a flow rate of 0.025 mL/min, the spinneret diameter 0.6 mm and the distance from tip to the collector plate (TCD) 16.5 cm. Each membrane was fabricated for about 3 h with a thickness ranging from 40 to 60 µm which were measured from the cross-section of the membrane using an optical microscope (OM, Olympus BX53M).

Characterization

The morphologies of AV/PVA nanofiber membranes were characterized by scanning electron microscopy (SEM, TESCAN VEGA3 LMU). Before inserting in the specimen chamber, a surface of the membrane specimen was metallic coated with Au-Pd alloy. Fiber diameter observed from two different regions of the membrane were measured using the ImageJ software. The AV-PVA membranes were tensile tested using a testing machine (Zwick Z0.5 Germany) at a crosshead speed 10 mm/min and a gauge length 20 mm, in which the specimens were prepared according to ASTM 882. The Equation determined tensile properties (tensile strength, tensile strain, and modulus elasticity) of the membranes is described elsewhere [5].

RESULTS AND DISCUSSION

Morphology

SEM micrographs for all produced electrospun AV-PVA nanofibers (AV-PVA nanofiber membranes) (Figure 2) show the un-beaded fibrous structure with relatively homogeneous fiber size distribution. AV concentration of electrospinning solution had a considerable effect on the morphology of nanofibers. Fibers at 2% and 4% AV concentration (Figure 2(b) and 2(c)) appears slightly smaller than that at neat PVA (0%) (Figure 2(a)) and 6% AV concentration (Figure 2(d)).

As a confirmation to ensure the observation of surface structure on the SEM images (Figure 2), the diameter distribution of the electrospun AV-PVA nanofibers at each AV concentration is shown in Figure 3. Almost 50% of the fiber diameter (400-500 nm) formed in a neat PVA membrane (0% AV concentration) had the average fiber diameter of 377 ± 1.1 nm (Figure3a). AV-PVA membranes with 2% and 4% AV concentration show the near similar fiber distribution in the range between 300 and 400 nm. Those mean fiber diameters were 342 ± 0.8 nm and 337 0.6 nm, respectively. The increase of AV concentration to 6% significantly increased mean fiber diameter to 810 ±3.4 nm (Figure 3(d)). The addition of AV concentration (2% and 4%) in PVA solution reduced the fiber diameter, but not with that of 6% AV concentration. This result has a similar trend to the other studies [10, 11].

Abdullah *et al*. [10] who have also studied the nanofiber membrane of AV-PVA showed that the average fiber diameter of AV-PVA 5% (w/v) (123 nm) is smaller than that of neat PVA (168 nm). The trend that insertion of AV into PVA solution reduced the fiber size is consistent with this study, although those average fiber diameters were much smaller compared to the present results: mean diameter of fiber at 0% and (2% and 4%) AV concentrations as described above. In addition, electrospun AV/PVA/PVP/PEG nanofiber with AV concentration 1%, 2% and 3% produced the fiber size higher than 500 nm for all concentrations [11]. Those

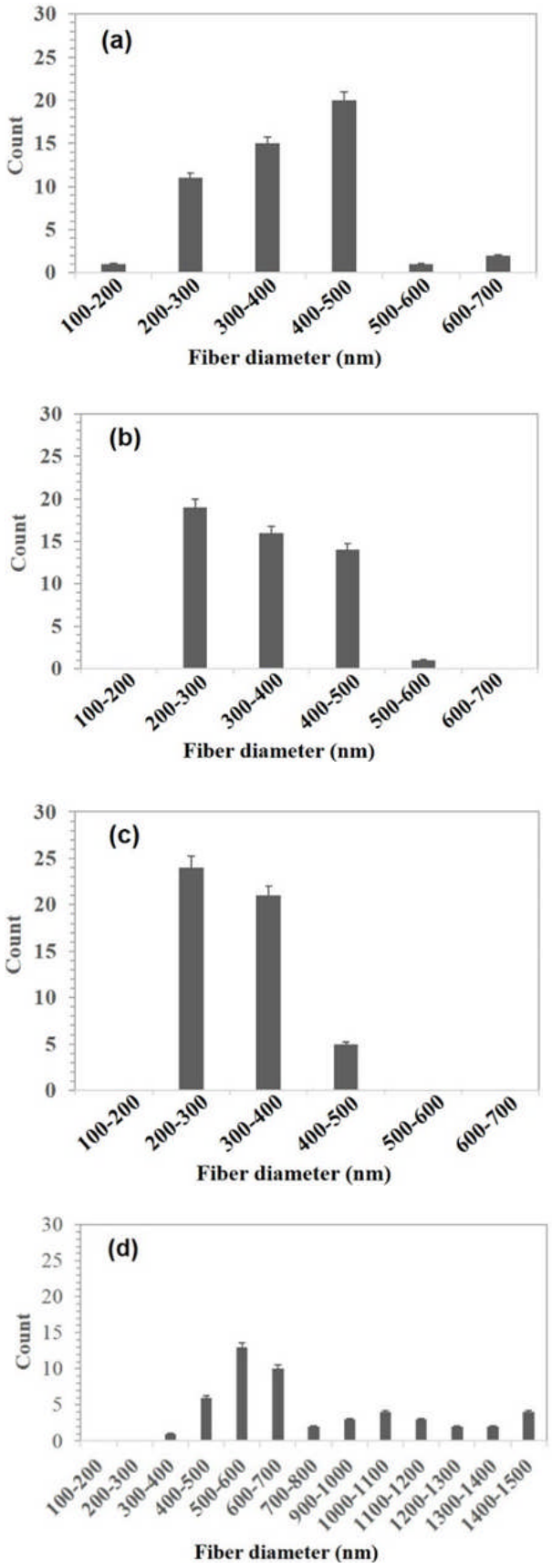

Figure 3. (a), (b), (c) and (d) are the corresponding distribution of fiber diameter formed in SEM images Figure 4(a), 4b, 4c and 4d, respectively.

sizes were higher than the present results at AV concentration of 2% and 4%, but near to that at 6% AV concentration. Except by the reason due to a different kind of used polymers, viscosity of the polymer solution (electrospinning solution) and also different operating condition during electrospinning might be the cause of those different results.

The morphology of fibers can be altered by changing the viscosity of the solution. A solution with low viscosity would have a low viscoelastic and columbic repulsion forces, causing the jet stream accessible to break-up and generate the beaded fiber structure. Inversely, the high viscosity of the solution leads to too large viscoelastic and the jet stream difficult to break-up. This condition would produce relatively large fiber diameter [15].

The morphology of fiber illustrated in all AV-PVA nanofiber membranes (Figure 2) with fiber size distribution displayed in Figure 3 is attributed to the viscosity of AV-PVA solution at the different AV concentrations (Table 1). An increase of AV concentration increased the viscosity of the AV-PVA solutions. The viscosity of AV-PVA solution at different AV concentrations used in this study is included in the range of 1 - 20 poise as reported by Amariei *et al*. [15] who have indicated that generally a spinning solution with a viscosity of those range values is ascribed to be a proper solution for electrospinning. As a result, the morphology of fibers resulted from blended AV and PVA in this study is closely associated with the viscosity of the spinning solution: i.e. as the viscosity increases the fiber size becomes larger. The addition of AV at the concentration ranging from 2% to 6% resulted in bead-free fiber structures. The typical structure obtained from the present work may lead to increased porosity which would be applicable for wound dressing material [11].

Table 1. Viscosity of AV/PVA solution at different AV concentration.

Specimen	Concentration % (w/v)	Viscosity (cP)
Neat PVA	0	539.9
AV-PVA-2	2	959.8
AV-PVA-4	4	999.8
AV-PVA-6	6	1015.0

Tensile Properties

As mentioned in the previous section, that the nanofiber membrane used for wound dressing should behave high mechanical properties such as high tensile strength, but low stiffness for flexibility. Herein, the tensile properties of the produced AV-PVA nanofiber membranes with different AV concentrations were evaluated as shown in Figure 4. Tensile strength and tensile modulus of AV-PVA nanofiber membrane gradually increase at AV concentration 2% and 4% and drastically decreases at a concentration of 6% (w/v). Alteration of

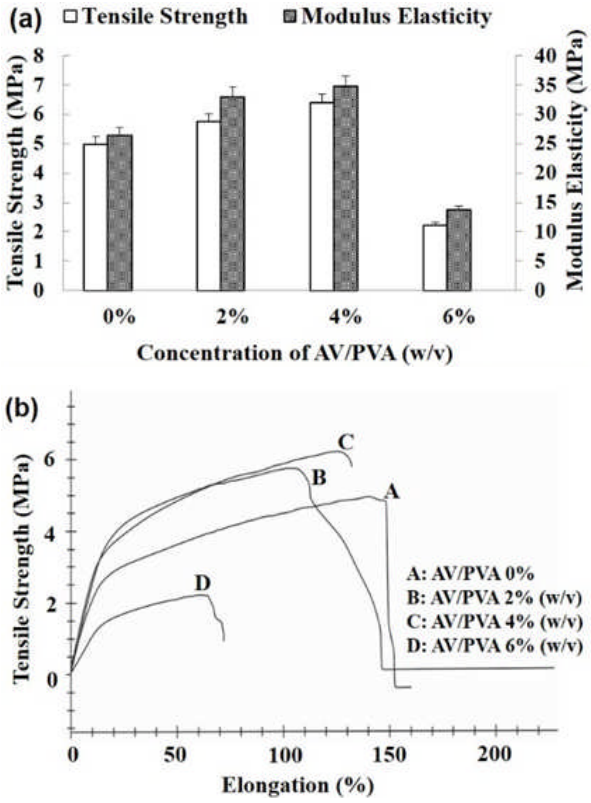

Figure 4. (a). Tensile strength and modulus versus concentration of AV/PVA, and (b). tensile strength versus elongation at break.

the tensile properties could be related to the formed fiber morphology due to different viscosity of AV-PVA solution. In this case, the membrane at AV concentration of 4% having the smallest mean fiber diameter shows the highest tensile strength and modulus. As the fiber diameter is going lower, a surface area of the fiber per unit volume enhances and interaction point between individual fibers also increases. These lead to an expanding fiber network that provides an impact on higher tensile strength and modulus.

In this case, an increase of tensile strength and modulus is also considered by uniformly blended and strong interfacial interaction between AV and PVA, in which the highest value obtained at 4% AV concentration. Besides, an increase and decrease of the mechanical properties of the AV-PVA nanofiber membranes can be attributed to the degree of crystallinity of the membrane that was studied by differential scanning calorimetry (DSC) on poly lactic acid (PLA)/epoxidized palm olein (EPO) blends [16]. A degree of crystallinity can be estimated according to the enthalpy obtained from DSC curves. Related to the present results, PVA is semi-crystalline. The addition of AV concentration from 0% to 4% may enhance the degree of crystallinity, leading to improving the tensile strength and modulus. However, at a concentration higher than 4% may gradually alter a crystalline to be amorphous and lowered the tensile strength and modulus.

In addition, experimental data on the correlation of tensile strength and elongation at break (Figure 4b) displayed that elongation at break of the membrane at 0% AV concentration reveals the highest, indicating high mobility of PVA chains and high flexibility of neat PVA membrane [16]. The elasticity of the membrane decreases with an increase in AV concentration.

The present result has specified that AV-PVA membranes at 2% and 4% AV concentrations are higher tensile properties than that at 0% and 6% AV concentrations: i.e. tensile strength (5.74 and 6.38 MPa) and tensile modulus (33.99 and 34.75 MPa) for 2% and 4%, respectively are higher than tensile strength (4.98 and 2.22 MPa), tensile modulus (26.45 and 13.69 MPa) for 0% and 6%, respectively. As mentioned in the previous Section, the properties indicated by the native skin are the tensile strength (5.00 - 30.00 MPa), Young modulus (4.6 - 20.0 MPa) and elongation to break (35.00 - 115.00%) [7]. Based on those results, the tensile strength of the present membrane at 2% and 4% AV concentration is close to that of the native skin, but Young modulus (tensile modulus) and elongation at break are slightly higher than that of the natural skin. Further research should be carried out to obtain the tensile properties of electrospun AV-PVA nanofiber correctly included in the range of those of the native skin.

CONCLUSIONS

The AV-PVA nanofiber membranes have been successfully fabricated with some following properties. All produced membranes showed beads-free fibrous structure. Insertion AV at the concentration of 2% and 4% reduced the fiber size. Tensile properties of the membranes at AV concentration of 2% and 4% were higher than that of 0% and 6%. Those tensile strengths (5.74 and 6.38 MPa) are included in the range of that indicated by the natural skin. Tensile modulus (33.99 and 34.75 MPa) and elongation at break (135% and 145%), respectively, however, were higher than that of the natural skin.

The result has pointed out that the best tensile properties and fiber morphologies obtained by AV-PVA nanofiber membrane at the concentration ranging from 2% to 4%. This study has verified that the concentration and viscosity of AV-PVA solution acted as a vital role in affecting the fiber morphology and the tensile properties of produced AV-PVA membranes.

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